

Revision Log

<i>Revision No.</i>	<i>Effective Date</i>	<i>Prepared By</i>	<i>Description of Changes</i>	<i>Affected Pages</i>
R0	04/26/00	Bart Vanden Plas	Initial procedure.	All
R1	05/27/03	Keith Greene	Rewritten to streamline and update process.	All
Review	04/20/2004	Nita Patel	Deemed process adequate.	All

Routine Validation of Semivolatile Organic Data

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Routine Validation of Semivolatile Organic Data

1.0 PURPOSE

- 1.1 This standard operating procedure (SOP) represents the minimum standards for evaluating routine semivolatile organic compound (SVOC) analytical data. These data can be generated for the Los Alamos National Laboratory (LANL), Risk Reduction and Environmental Stewardship—Remediation (RRES-R) Program using SW-846 Method 8270 or the comparable Contract Laboratory Program (CLP) methods under the current statement of work (SOW) for analytical services. The evaluation of data by this procedure is not specific to a particular data use, although this procedure may be used to develop focused requirements specific to a particular data use.
- 1.2 Implementation of this procedure results in a tabulation of data compliances and noncompliances identified relative to expectations for data quality based on national guidelines for data review (U.S. Environmental Protection Agency [EPA] 1999, 66649). Data noncompliance is noted through the application of qualifier flags (Attachment A) and reason codes (Attachment B) to the reported results. Because the acceptance criteria used for this procedure are not based on site-specific acceptance criteria, the results of this validation procedure are intended to be used as general indicators of data quality and should not be construed as a definitive identification of data usability.
- 1.3 Nothing in this SOP precludes the validator from going beyond the minimum requirements specified in this SOP. To address data quality issues in a data package, the validator may assign qualifiers based on his or her professional judgment. Implementation of this procedure also may be followed by a more focused and data-use-specific evaluation of data, especially if implementation of this SOP indicates that the data may contain technical deficiencies. The validator will note any need for a more focused validation on the Data-Validation Cover Sheet (Attachment C). The validator will use the SVOC Data-Validation Checklist (Attachment D) to record the specific validation steps conducted.

2.0 SCOPE

- 2.1 All **RRES-R Personnel** shall implement this mandatory SOP who evaluate routine SVOC analytical data.
- 2.2 **Subcontractors** performing work under the RRES-R Program's quality program shall follow this SOP.

3.0 TRAINING

- 3.1 **RRES-R Personnel** shall train to and use the current version of this SOP; contact the author if the SOP text is unclear.
- 3.2 **RRES-R Personnel** using this SOP shall document training in the RRES-R training database located at <http://erinternal.lanl.gov/Training/login.asp> in accordance with QP-2.2.
- 3.3 The responsible **supervisor** shall monitor the proper implementation of this procedure and ensure that the appropriate personnel complete all applicable training assignments.
- 3.4 All **data validators** implementing this SOP shall possess a minimum of a bachelors degree in chemistry or one of the physical sciences AND either two years experience in generating analytical data in an environmental analytical laboratory or two years data-validation experience.
- 3.5 Experienced RRES-R **validators** shall directly supervisor inexperienced validators, applying review signatures on ten data record packages, indicating satisfactory validation.
- 3.6 RRES-R **validators** shall demonstrate familiarity with the EPA national functional guidelines for data review.

4.0 DEFINITIONS

- 4.1 *Analyte*—The element, nuclide, or ion that a chemical analysis seeks to identify and/or quantify; the chemical constituent of interest.
- 4.2 *Area count*—Integrated area under a chromatographic peak. The area count is proportional to the amount of compound present in the aliquot introduced into the chromatograph.
- 4.3 *Continuing calibration verification (CCV)*—Check standards used to determine if the instrument response to analyte concentration is within acceptable bounds relative to the initial calibration. A CCV is performed every 12 h of operation or (for inorganic and high explosives [HEs]) every 10 injections (samples and/or quality control [QC] samples), whichever is more frequent, thus verifying the satisfactory performance

of an instrument on a day-to-day basis. The continuing calibration 12-h period assumes that the instrument has not been shut down since the initial calibration.

- 4.4 *Data validator*—Person who has met the minimum standards of training established by the RRES-R Program for data validation and who performs data validation on behalf of the RRES-R Program (hereinafter referred to as the “validator”).
- 4.5 *Detect (inorganic and organic)*—Sample result above the method detection limit (MDL) as reported by the contract analytical laboratory. The contract laboratory reports the concentration of the analyte in the sample.
- 4.6 *Form 1*—Organic analysis data sheet for each individual sample that includes the sample information needed to identify the sample and the analytical results for the sample. See the SOW for analytical services (Records Processing Facility [RFP] No. 9-XS1-Q4257) for a more complete definition.
- 4.7 *Holding time*—Maximum elapse of time that a sample can be stored without unacceptable changes in analyte concentrations. Holding times apply under prescribed conditions, and deviations from these conditions may affect the holding time. Extraction holding time refers to the time lapse from sample collection to sample preparation; analytical holding time refers to the time lapse between sample preparation and analysis.
- 4.8 *Initial calibration*—Process used to establish the relationship between instrument response and analyte concentration at several analyte-concentration values to demonstrate that an instrument is capable of acceptable analytical performance.
- 4.9 *Instrument performance check*—Analysis of a chemical of known relative mass abundances that indicates how well a mass spectrometer is calibrated.
- 4.10 *Internal standard (IS)*—Chemical compound added to every blank, sample, and standard extract at a known concentration that is used to (1) compensate for analyte concentration changes that might occur during storage of the extract and (2) compensate for quantitation variations that can occur during analysis. ISs are used as the basis for quantitation of target analytes.
- 4.11 *Laboratory control sample (LCS)*—Known matrix that has been spiked with compound(s) representative of the target analytes. The LCS is used to document laboratory performance. The acceptance criteria for LCSs are method specific.

- 4.12 *Laboratory duplicate sample*—Portions of a sample taken from the same sample container, prepared for analysis and analyzed independently but under identical conditions; used to assess or demonstrate acceptable laboratory method precision at the time of analysis. Each duplicate sample is equally representative of the original material. Duplicate analyses also are performed to generate data, and to determine the long-term precision of an analytical method on various matrices.
- 4.13 *Laboratory qualifier (or laboratory flag)*—Codes applied to the data by the contract analytical laboratory to indicate, on a gross scale, a verifiable or potential data deficiency. These flags are applied using the EPA CLP guidelines (EPA 1994, 48639; EPA 1999, 66649).
- 4.14 *LANL data-validation qualifiers*—Data qualifiers defined by LANL and used in the RRES-R Program routine validation process. Attachment A lists all data qualifiers that are applicable to all analytical suites.
- 4.15 *LANL data-validation reason codes*—Codes applied to the sample data by data validators who are independent of the contract laboratory that performed the sample analysis. Reason codes provide an in-depth and analysis-specific explanation for applying the qualifier along with a description of the potential impact on the data use. For a complete list of data qualifiers applicable to any particular analytical suite, consult the appropriate RRES-R Program SOP.
- 4.16 *Lower acceptance limit (LAL)*—Lowest limit that is acceptable, based on the quality control (QC) criteria for a specific QC sample for a specific method. Any results lower than the LAL are qualified following this routine validation procedure.
- 4.17 *Matrix spike*—An aliquot of sample spiked with a known concentration of target analyte(s). Matrix-spike samples are used to measure the ability to recover prescribed analytes from a native sample matrix. Spiking typically occurs before sample preparation and analysis.
- 4.18 *Method blank*—Analyte-free matrix to which all reagents are added in the same volumes or proportions as those used in the environmental sample processing, and which is prepared and analyzed in the same manner as the corresponding environmental samples. A method blank is used to assess the potential for sample contamination during preparation and analysis.
- 4.19 *Method detection limit (MDL)*—Minimum concentration of a substance that can be measured and reported with known statistical confidence that the analyte concentration is greater than zero. The MDL is determined by analyzing samples of a given matrix type that contain the

analyte after the sample is subjected to the usual preparation and analyses. The MDL is used to establish detection status.

- 4.20 *Nondetect (organics)*—Sample result that is less than the MDL. The laboratory reports nondetects as undetected at the reporting limit (RL).
- 4.21 *Percent difference %D*—Measure of deviation from the initial calibration to the continuing calibration, based on calibration factors.
- 4.22 *Percent recovery (%R)*—Amount of material detected in a sample (minus any amount already in the sample) divided by the amount added to the sample and expressed as a percentage.
- 4.23 *Percent relative standard deviation (%RSD)*—Evaluation of deviation between the concentrations versus analyte response over the dynamic linear calibration range. The basic equation is $\%RSD = (\text{Std dev}/\text{av}) \bullet 100$.
- 4.24 *Relative response factor (RRF)*—Relationship between analyte concentrations versus area response.
- 4.25 *Reporting limit (RL)*—Lowest concentration that reliably can be achieved within specified limits of precision and accuracy during routine analytical-laboratory operating conditions. The low point on a calibration curve should reflect this reporting limit. The RL is not used to establish detection status.
- 4.26 *Request number (RN)*—An identifying number assigned by the RRES-R Program to a group of samples that are submitted for analysis.
- 4.27 *Routine data*—Data generated using analytical methods that are identified as routine methods in the current RRES-R Program SOW for analytical services.
- 4.28 *Routine data validation*—Process of reviewing analytical data relative to quantitative routine acceptance criteria. The objective of routine data validation is two-fold: (1) to estimate the technical quality of the data relative to minimum national guidelines adopted by the RRES-R Program; and (2) to indicate to data users the technical data quality at a general level by assigning qualifier flags to environmental data whose quality indicators do not meet acceptance criteria.
- 4.29 *Surrogate compound (surrogate)*—Organic chemical compound used in the analyses of organic target analytes that is similar in composition and behavior to target analytes but is not normally found in environmental samples. Surrogates are added to every blank, sample, and spike to evaluate the efficiency with which analytes are recovered during extraction and analysis.

- 4.30 *Target analyte*—An element, chemical, or parameter, the concentration, mass, or magnitude of which is designed to be quantified by use of a particular test method.
- 4.31 *Tentatively identified compound (TIC)*—Chemical compound detected in a sample that is not a target analyte, IS, or surrogate compound. Up to 30 chromatographic peaks may be subject to mass spectral matching for identification as TICs. Tentative identification is based on comparison of the compound mass spectrum to an industry-standard mass-spectra library using both a statistical matching algorithm and the professional judgment of the analyst.
- 4.32 *Upper acceptance limit (UAL)*—Highest limit that is acceptable, based on the QC criteria for a specific QC sample for a specific method. All results greater than the UAL are qualified following this routine validation procedure.

5.0 RESPONSIBLE PERSONNEL

The following personnel are responsible for activities identified in this procedure:

- Data validator (see definition 4.4)
- RRES-R Personnel
- Project Team Leader
- Quality Program Project Leader
- Supervisor
- User

6.0 PROCEDURE

Data Validators shall perform the following work processes, and shall make any deviations from this SOP in accordance with QP-5.7 and/or SOP-01.01.

6.1 Preparing for Data Validation

1. Obtain the required current versions of the SVOC Data-Validation Checklist form (Attachment D) from the RRES-R Program website <http://erinternal.lanl.gov/Quality/forms.htm>.
2. Obtain from the Sample Management Office (SMO) of the Field Support Facility (FSF), the data-record packages containing the sample data to be validated.

A. Prepare a Data-Validation Cover Sheet (Attachment C) by completing the top part of the cover sheet and placing a check next to the analytical suites for which the validation is being performed.

B. If any data are rejected, check the rejected box and notify the project chemist immediately.

Note: You may use a single cover sheet when validating multiple analytical suites under the same RN.

Note: Use a separate sheet of paper to document each deficiency identified beyond the extent of this procedure, including phone conversations with the analytical laboratory concerning these deficiencies. Attach these sheets to the Data-Validation Cover Sheet.

3. Verify that the following items are present in the data-record package:

- A signed LANL chain of custody (COC) record
- The result forms (CLP Form 1 or equivalent) for each sample
- The reconstructed ion chromatograms (RICs) for each sample
- The RICs for the standards
- The raw and background subtracted spectra (BSS) of the identified compounds
- The quantitation reports
- The QC forms (CLP 2A [Deuterated Monitoring Compound Recovery], 3A [Matrix Spike/Matrix Spike Duplicate Recovery], 4A [Method Blank Summary], 6A [gas chromatography mass spectrometry {GC/MS} Initial Calibration Data], 8A [Internal Standard Area and RT Summary], or the equivalent) for water and/or soils, as appropriate
- The TIC forms (CLP Form V-TIC, or equivalent) which are required only if the RRES-R Program requested the TIC reports
- The mass spectra of the TICs with the best library matches (required only if the RRES-R Program requested the TIC reports)

4.	IF the required documentation for the data-record package is...	FOR...	THEN...
	Complete,		<ul style="list-style-type: none"> Go to Step 6.
	Missing,	< 6 mo.	<ul style="list-style-type: none"> Contact the analytical laboratory and/or the SMO. Allow 3 business days for submittal. Go to Step 5.
	Missing,	= 6 mo.	<ul style="list-style-type: none"> Contact the analytical laboratory and/or the SMO. Allow 10 business days for submittal. Go to Step 5.

Note: To expedite the validation process, the validator may request that the contract laboratory forward the missing information by e-mail or fax directly to the validator within 24 h of notification.

5.	IF the analytical laboratory...	THEN...
	Submits the documentation within the specified time period,	<ul style="list-style-type: none"> Go to Step 6.
	Does <u>not</u> submit documentation within the specified time period,	<ul style="list-style-type: none"> Notify the SMO for contract-compliance action. Go to Step 6.

6. Record the presence or absence ("Yes" or "No") of each item, as appropriate, in the completeness check section of the Data-Validation Cover Sheet.
 - A. If the RRES-R Program did not request the TICs, record "n/a" in blocks 9 and 10 of the completeness checklist.
 - B. In the Data-Validation Cover Sheet (completeness check section), note any samples whose data are missing from the data-record package under comments/problems noted.

7. Photocopy the following items:

- The chain of custody form
- Form 1 from the analytical laboratory (used during the validation process)

Note: Do not record the data-validation qualifiers and the reason codes on the original form (Form 1).

Note: Each page of Form 1 must be initialed and dated by the validator; these initials and date must be present even if the validator accepts laboratory qualification.

Note: Submit the photocopies of the items listed in Step 7 as attachments to the completed Data-Validation Checklists.

8. Go to Section 6.2, "Verifying the Holding Time."

6.2 Verifying the Holding Time

Table 6.2-1
Holding Time Acceptance Criteria

Sample Matrix	Extraction Holding Time (days)	Analysis Holding Time (days)
Soil	14	40
Water	7	40
The current SOW for analytical services lists applicable storage conditions.		

1. IF...	THEN...
<u>All</u> the samples were extracted <u>and</u> analyzed within their holding times (Table 6.2-1),	<ul style="list-style-type: none">• Record "No" on lines 1, 2, and 3 of the SVOC Data-Validation Checklist (Attachment D).• Go to Section 6.3, "Verifying the Instrument Performance Check."
<u>Any</u> samples were <u>not</u> extracted within the holding time (Table 6.2-1),	<ul style="list-style-type: none">• Calculate the number of days that the holding time was exceeded.• Go to Step 2.

2.	IF any extraction holding time was exceeded by...	THEN...
	= 2 times the holding time acceptance criterion,	<ul style="list-style-type: none"> Record "Yes" on line 1 and "No" on line 2 of the SVOC Data-Validation Checklist. Qualify the detected analytes as estimated with a potential negative bias (J-, SV9) and the nondetected analytes as estimated (UJ, SV9) on Form 1. Go to Step 3.
	2 times the holding time acceptance criterion,	<ul style="list-style-type: none"> Record "Yes" on line 2 and "No" on line 1 of the SVOC Data-Validation Checklist. Qualify all the affected samples as rejected (R, SV9a) on the Form 1. Go to Step 3.

3.	IF the analytical holding time was...	THEN...
	<u>Not</u> exceeded,	<ul style="list-style-type: none"> Record "No" on line 3 of the SVOC Data-Validation Checklist. Go to Section 6.3, "Verifying the Instrument Performance Check."
	Exceeded,	<ul style="list-style-type: none"> Record "Yes" on line 3 on the SVOC Data-Validation Checklist. Qualify the affected analytes as rejected (R, SV9b) on Form 1. Go to Section 6.3, "Verifying the Instrument Performance Check."

Note: Samples have to be analyzed within 40 days after extraction to meet the analytical holding time acceptance criteria.

6.3 Verifying the Instrument Performance Check

IF the decafluorotriphenylphosphine (DFTPP) instrument performance check was...	THEN...
Completed within 12 h of the corresponding sample and calibration analyses <u>and</u> passed the acceptance criteria,	<ul style="list-style-type: none"> Record "No" on line 4 of the SVOC Data-Validation Checklist. Go to Section 6.4, "Verifying the Initial Calibration."
<u>Not</u> completed within 12 h of the corresponding sample analyses <u>or</u> failed the acceptance criteria,	<ul style="list-style-type: none"> Record "Yes" on line 4 of the SVOC Data-Validation Checklist. Qualify the affected analytes as rejected (R, SV16a) on the individual sample Form 1. Go to Section 6.4, "Verifying the Initial Calibration."

6.4 Verifying the Initial Calibration

1.	IF the initial calibration information is...	THEN...
	Present,	<ul style="list-style-type: none"> Record "No" on line 5 of the SVOC Data-Validation Checklist. Go to Step 2.
	Missing,	<ul style="list-style-type: none"> Record "Yes" on line 5 of the SVOC Data-Validation Checklist. Contact the analytical laboratory and the SMO to request the missing information (see Section 6.1-4). If the laboratory is unable to provide the missing information, qualify affected samples as rejected (R, SV16) on the individual sample Form 1.

2.	IF the initial calibration ...	THEN...
	Has five calibration points <u>and</u> a standard at or below the reporting limit (RL),	<ul style="list-style-type: none"> Record "No" on line 6 of the SVOC Data-Validation Checklist. Go to Step 3.
	Does <u>not</u> have either five calibrations points <u>or</u> a standard at or below the RL.	<ul style="list-style-type: none"> Record "Yes" on line 6 of the SVOC Data-Validation Checklist. Qualify the affected analytes as rejected (R, SV7) on the individual sample Form 1. Go to Step 3.

Note: If the detects are within the range of the acceptable calibration standards, the results may be qualified as estimated (J) with Reason Code SV7, based on the professional judgment of the validator.

3.	IF the percent relative standard (%RSD) for...	THEN...
	<u>Each</u> analyte is = 30%, <u>or</u> if the %RSD is not used, then the correlation coefficient is = 0.995.	<ul style="list-style-type: none"> Record "No" on line 7 of the SVOC Data-Validation Checklist. Go to Step 4.
	<u>Any</u> analyte is > 30% <u>or</u> if the correlation coefficient is < 0.995,	<ul style="list-style-type: none"> Record "Yes" on line 7 of the SVOC Data-Validation Checklist. Qualify the affected analytes as estimated (J, SV7a/UJ, SV7a) on the individual sample Form 1. Go to Step 4.

4.	IF the minimum relative response factor (RRF) is...	THEN...
	= 0.05 for each analyte,	<ul style="list-style-type: none"> Record "No" on line 8 of the SVOC Data-Validation Checklist. Go to Section 6.5, "Verifying the Continuing Calibration."

4. IF the minimum relative response factor (RRF) is...	THEN...
< 0.05 for any analyte,	<ul style="list-style-type: none"> Record "Yes" on line 8 on the SVOC Data-Validation Checklist. Qualify the detected analytes as estimated (J, SV7b) and the nondetected analytes as rejected (R, SV7b) on the individual sample Form 1. Go to Section 6.5, "Verifying the Continuing Calibration."

6.5 Verifying the Continuing Calibration

1. IF the continuing calibration information is...	THEN...
Present,	<ul style="list-style-type: none"> Record "No" on line 6 of the SVOC Data-Validation Checklist. Go to Step 2.
Missing,	<ul style="list-style-type: none"> Record "Yes" on line 9 of the SVOC Data-Validation Checklist. Contact the analytical laboratory and the SMO to request the missing information (see Section 6.1-4). If the laboratory is unable to provide the missing information, qualify all results as rejected (R, SV16) on the individual sample Form 1. Go to Step 2.

2. IF the percent difference (%D) for...	THEN...
<u>Each</u> analyte is = 25%,	<ul style="list-style-type: none"> Record "No" on line 10 of the SVOC Data-Validation Checklist. Go to Step 3.

2.	IF the percent difference (%D) for...	THEN...
	<u>Any</u> analyte is > 25%,	<ul style="list-style-type: none"> Record "Yes" on line 10 of the SVOC Data-Validation Checklist and Qualify the affected analytes as estimated (J, SV7a/UJ, SV7a) on the individual sample Form 1. Go to Step 3.
3.	IF the minimum RRF is...	THEN...
	= 0.05 for <u>each</u> analyte,	<ul style="list-style-type: none"> Record "No" on line 11 of the SVOC Data-Validation Checklist. Go to Section 6.6, "Verifying the Method-Blank Results."
	< 0.05 for <u>any</u> analyte,	<ul style="list-style-type: none"> Record "Yes" on line 11 on the SVOC Data-Validation Checklist. Qualify the detected analytes as estimated (J, SV7b) and the nondetected analytes as rejected (R, SV7b) on the individual sample Form 1. Go to Section 6.6, "Verifying the Method-Blank Results."

6.6 Verifying the Method-Blank Results

Note: The data validator must compare method-blank results to the contractually required estimated quantitation limits (EQLs) for each analytical batch.

1.	IF the method blank information is...	THEN...
	Present,	<ul style="list-style-type: none"> Record "No" on line 12 of the SVOC Data-Validation Checklist. Go to Step 2
	Missing,	<ul style="list-style-type: none"> Record "Yes" in line 12 of the SVOC Data-Validation Checklist. Contact the analytical laboratory and the SMO to request the missing information (see Section

1. IF the method blank information is...	THEN...
	6.1-4). <ul style="list-style-type: none"> • If the laboratory is unable to provide the missing information, qualify all results as rejected (R, SV4b) on the individual sample Form 1. • Go to Step 2.
2. IF the method blank has...	THEN...
<u>No</u> contamination,	<ul style="list-style-type: none"> • Record "No" on lines 13 and 14 of the SVOC Data-Validation Checklist. • Go to Section 6.7, "Verifying the Internal Standards."
Contamination,	<ul style="list-style-type: none"> • Go to Step 3.
3. IF the concentrate of any analyte in a sample is...	THEN...
= 5 times the concentration of that analyte in the corresponding method blank (10 times for common laboratory contaminants),	<ul style="list-style-type: none"> • Record "Yes" on line 13 of the SVOC Data-Validation Checklist. • Qualify the detected analytes as undetected (U, SV4) on the individual sample Form 1. • Go to Section 6.7, "Verifying the Internal Standards."
> 5 times the concentration of that analyte in the corresponding method blank (10 times for common laboratory contaminants),	<ul style="list-style-type: none"> • Record "Yes" on line 14 of the SVOC Data-Validation Checklist. • Qualify the detected analytes as estimated (J, SV4a) on the individual sample Form 1. • Go to Section 6.7, "Verifying the Internal Standards."

Note: Check the concentrations of common laboratory contaminants in the diluted samples. If the concentration is within 10 times the concentration of the blank divided by the dilution factor, and the sample was diluted for compounds other than the common

laboratory contaminant in question, use professional judgment to apply the qualification criteria listed in this section.

6.7 Verifying the Internal Standards

Note: The internal standard (IS) percent recoveries (%Rs) are calculated based on the area counts compared to the area counts in the applicable CCV. The percent recovery (%R) acceptance limits are that the area counts must be within a factor of 2 when compared to the area counts in the applicable CCV. This translates to an acceptable range of greater than or equal to 50%R and less than or equal to 200%R.

**Table 6.7-1
RRES-R Program-Required SVOC Internal Standards**

IS No.	IS Name	IS No.	IS Name
1	1,4-dichlorobenzene-d4	4	Phenanthrene-d10
2	Naphthalene-d8	5	Chrysene-d12
3	Acenaphthalene-d10	6	Perylene-d12

1.	IF the IS information is...	THEN...
	Present for the six required ISs (Table 6.7-1),	<ul style="list-style-type: none"> Record "No" on line 15 of the SVOC Data-Validation Checklist. Go to Step 2.
	Missing,	<ul style="list-style-type: none"> Record "Yes" on line 15 of the SVOC Data-Validation Checklist. Contact the analytical laboratory and the SMO to request the missing information. If the laboratory is unable to provide the missing information, qualify all results as rejected (R, SV2a) on the individual sample Form 1. Go to Step 2.

2.	IF the IS areas for...	THEN...
	<u>All</u> of the samples are = 50%R <u>and</u> = 200%R of the previous 12-h CCV,	<ul style="list-style-type: none"> Record "No" on line 16 of the SVOC Data-Validation Checklist Go to Step 3.
	<u>Any</u> of the IS compounds in any sample are < 50%R <u>or</u> > 200%R,	<ul style="list-style-type: none"> Record "Yes" on line 16 of the SVOC Data-Validation Checklist. Qualify analytes that are quantitated against the IS in question as estimated (J, SV1/UJ, SV1) on the individual sample Form 1. Go to Step 3.

3.	IF the IS retention times for...	THEN...
	<u>All</u> of the IS compounds in all samples <u>have not</u> shifted by more than 30 s from the applicable CCV,	<ul style="list-style-type: none"> Record "No" on line 17 of the SVOC Data-Validation Checklist. Go to Step 4.
	<u>Any</u> of the IS compounds in any sample <u>have</u> shifted by more than 30 s from the previous 12-h CCV,	<ul style="list-style-type: none"> Record "Yes" on line 17 of the SVOC Data-Validation Checklist. Qualify the detected analytes as estimated (J, SV0) and the nondetected analytes as rejected (R, SV0) on the individual sample Form 1. Go to Step 4.

Note: If the mass spectrum does not support the reported detection, consider the analyte as possibly undetected and reject the results (R, SV0) rather than using U, SV8.

4.	IF the IS areas for any of the IS compounds in a sample are...	THEN...
	= 200%R,	<ul style="list-style-type: none"> Record "No" on line 18 of the SVOC Data-Validation Checklist. Go to Step 5.

4. IF the IS areas for any of the IS compounds in a sample are...	THEN...
> 200%R,	<ul style="list-style-type: none"> • Record "Yes" on line 18 of the SVOC Data-Validation Checklist. • Qualify all the detected analytes quantitated against the IS in question as estimated (J, SV1b) on the individual sample Form 1. • Go to Step 5.

5. IF the IS areas for...	THEN...
<u>All</u> of the IS compounds in <u>all</u> samples show = 50%R,	<ul style="list-style-type: none"> • Record "No" on lines 19 and 20 of the SVOC Data-Validation Checklist. • Go to Section 6.8, "Verifying the Surrogate Recoveries."
<u>Any</u> of the IS compounds in <u>any</u> sample show < 50%R but = 10%R,	<ul style="list-style-type: none"> • Record "Yes" on line 19 of the SVOC Data-Validation Checklist. • Qualify the analytes quantitated against the IS in question as estimated (J, SV1a/UJ, SV1a) on the individual sample Form 1. • Go to Step 6.

6. IF the IS area for...	THEN...
<u>All</u> of the IS compounds in <u>all</u> samples show = 10%R,	<ul style="list-style-type: none"> • Record "No" on line 20 of the SVOC Data-Validation Checklist. • Go to Section 6.8, "Verifying the Surrogate Recoveries."

6. IF the IS area for...	THEN...
<u>Any</u> of the IS compounds in <u>any</u> sample show < 10%R,	<ul style="list-style-type: none"> Record "Yes" on line 20 of the SVOC Data-Validation Checklist. Qualify the detected analytes as estimated (J, SV2) and the nondetected analytes as rejected (R, SV2) for analytes that are quantitated against the IS in question on the individual sample Form 1. Go to Step 6.8, "Verifying the Surrogate Recoveries."

6.8 Verifying the Surrogate Recoveries

Note: The surrogate %R values that are outside the acceptance range listed in Table 6.8-1, as a result of sample dilution used to render target analytes quantifiable, are not subject to the validation-acceptance criteria presented in this section.

Table 6.8-1
SVOC Surrogates and Recovery Acceptance Ranges

Surrogate (Fraction)	Soil Matrix Acceptance Range (%R)	Water Matrix Acceptance Range (%R)
Nitrobenzene-d5–(base/neutral)	23–120	35–114
2-fluorobiphenyl–(base/neutral)	30–115	43–116
p-terphenyl-d14–(base/neutral)	18–137	33–141
phenol-d6–(acid)	24–113	10–110
2-fluorophenol–(acid)	25–121	21–110
2,4,6-tribromophenol–(acid)	19–122	10–123

1.	IF the surrogate information is...	THEN...
	Present,	<ul style="list-style-type: none"> Record "No" in line 21 of the SVOC Data-Validation Checklist. Go to Step 2.
	Missing,	<ul style="list-style-type: none"> Record "Yes" on line 21 of the SVOC Data-Validation Checklist. Contact the analytical laboratory and the SMO to request the missing information (see Section 6.1-4). If the laboratory is unable to provide the missing information, qualify all results as rejected (R, SV3f) on the individual sample Form 1. Go to Step 2.

2.	IF...	THEN...
	< two surrogate %Rs in each fraction in each sample are > the UAL,	<ul style="list-style-type: none"> Record "No" on line 22 of the SVOC Data-Validation Checklist Go to Step 3.
	two or more surrogate %R in any fraction for any sample are > the UAL,	<ul style="list-style-type: none"> Record "Yes" on line 22 of the SVOC Data-Validation Checklist. Qualify all positive analytes as estimated with a potential positive bias (J+, SV3) on the individual sample Form 1. Go to Step 3.

3.	IF...	THEN...
	< two surrogate %R in <u>each</u> fraction in <u>each</u> sample are < lower acceptance limit (LAL) but = to 10%,	<ul style="list-style-type: none"> Record "No" on line 23 of the SVOC Data-Validation Checklist. Go to Step 4.

3. IF...	THEN...
At least two surrogate %R in <u>any</u> fraction for <u>any</u> sample are < LAL but = to 10%,	<ul style="list-style-type: none"> Record "Yes" on line 23 of the SVOC Data-Validation Checklist. For the affected fraction, qualify detected analytes as estimated with a potential negative bias (J-, SV3a) and nondetected analytes as estimated (UJ, SV3c) on the individual sample Form 1. Go to Step 4.
4. IF...	THEN...
No surrogate %R in any sample is < 10%,	<ul style="list-style-type: none"> Record "No" on line 24 of the SVOC Data-Validation Checklist Go to Step 5.
Any surrogate %R in any sample is < 10%,	<ul style="list-style-type: none"> Record "Yes" on line 24 of the SVOC Data-Validation Checklist. Qualify the detected analytes in the affected fraction as estimated with a potential negative bias (J-, SV3b) and the nondetected analytes in the affected fraction as rejected (R, SV3d) on the individual sample Form 1. Go to Step 5.
5. IF there are...	THEN...
No fractions in any sample where one surrogate %R is < LAL and another surrogate %R is > UAL,	<ul style="list-style-type: none"> Record "No" on line 25 of the SVOC Data-Validation Checklist. Go to Section 6.9, "Verifying the Laboratory Control Sample Recoveries."

5. IF there are...	THEN...
Any fractions in any sample where one surrogate %R is < LAL and another surrogate %R is > UAL,	<ul style="list-style-type: none"> Record "Yes" on line 25 of the SVOC Data-Validation Checklist Qualify the affected analytes in the affected fraction as estimated (J, SV3e/UJ, SV3e) on the individual sample Form 1. Go to Section 6.9, "Verifying the Laboratory Control Sample Recoveries."

6.9 Verifying the Laboratory Control Sample Recoveries

Note: The laboratory may perform either a full list laboratory control sample (LCS) or a short spike list (CLP matrix spike list). For a full list LCS, qualify only those analytes that fail the LCS control limits. For shorter list LCSs, use professional judgment to determine which analytes to qualify when the LCS limits are not met.

1. IF the LCS information is...	THEN...
Present,	<ul style="list-style-type: none"> Record "No" on line 26 of the SVOC Data-Validation Checklist. Go to Step 2.
Missing,	<ul style="list-style-type: none"> Record "Yes" on line 26 of the SVOC Data-Validation Checklist. Contact the analytical laboratory and the SMO to request the missing information (see Section 6.1-4). If the laboratory cannot provide the missing information, qualify the affected samples as rejected (R, SV12) on the individual sample Form 1. Go to Step 2.

2. IF...	THEN...
No LCS analyte %R is > UAL,	<ul style="list-style-type: none"> Record "No" on line 27 of the SVOC Data-Validation Checklist. Go to Step 3.

2. IF...	THEN...
<u>Any</u> LCS analyte %R is > UAL,	<ul style="list-style-type: none"> Record "Yes" on line 27 of the SVOC Data-Validation Checklist. Qualify all the positive analytes as estimated with a potential positive bias (J+, SV12d) on the individual sample Form 1. Go to Step 3.
3. IF...	THEN...
<u>No</u> LCS analyte %R is < LAL but = to 10%,	<ul style="list-style-type: none"> Record "No" on line 28 of the SVOC Data-Validation Checklist. Go to Step 4.
<u>Any</u> LCS analyte %R is < LAL but = to 10%,	<ul style="list-style-type: none"> Record "Yes" on line 28 of the SVOC Data-Validation Checklist. Qualify the detected analytes as estimated with a potential negative bias (J-, SV12b) and the nondetected analytes as estimated (UJ, SV12c) on the individual sample Form 1.
4. IF...	THEN...
<u>No</u> LCS analyte %R is < 10%,	<ul style="list-style-type: none"> Record "No" on line 29 of the SVOC Data-Validation Checklist. Go to Section 6.10, "Verifying the Mass Spectra."
<u>Any</u> LCS analyte %R is < 10%,	<ul style="list-style-type: none"> Record "Yes" on line 29 of the SVOC Data-Validation Checklist. Qualify the detected analytes as estimated with a potential negative bias (J-, SV12a) and the nondetected analytes as rejected (R, SV12a) on the individual sample Form 1. Go to Section 6.10, "Verifying the Mass Spectra."

6.10 Verifying the Mass Spectra

1. IF...	THEN...
<u>All</u> of the required mass spectra are <u>present</u> ,	<ul style="list-style-type: none"> Record "No" on line 30 of the SVOC Data-Validation Checklist. Go to Step 2.
<u>Any</u> of the required mass spectra are <u>missing</u> ,	<ul style="list-style-type: none"> Record "Yes" on line 30 of the SVOC Data-Validation Checklist. Contact the analytical laboratory and the SMO to request the missing information (see Section 6.1-4). If the laboratory is unable to provide the missing information, qualify all results as rejected (R, SV8a) on the individual sample Form 1. Go to Step 2.

2. IF...	THEN...
<u>All</u> of the mass spectra meet the method specifications,	<ul style="list-style-type: none"> Record "No" on line 31 of the SVOC Data-Validation Checklist. Go to Section 6.11, "Verifying the Tentatively Identified Compounds."
<u>Any</u> of the mass spectrum do <u>not</u> meet the method specifications,	<ul style="list-style-type: none"> Record "Yes" on line 31 of the SVOC Data-Validation Checklist. Qualify detected analytes as undetected (U, SV8) on the individual sample Form 1. Go to Section 6.11, "Verifying the Tentatively Identified Compounds."

6.11 Verifying the Tentatively Identified Compounds

Note: If the order code contains "N" as the last letter, the "N" indicates that TICs were not requested.

IF the TIC information...	THEN...
Is <u>present</u> or was not requested,	<ul style="list-style-type: none"> • Check “No” on line 32 of the SVOC Data-Validation Checklist if the TICs are present. • Check “n/a” if TICs were not requested. • Go to Section 6.12, “Verifying the Dilutions.”
Was requested but is <u>missing</u> ,	<ul style="list-style-type: none"> • Record “Yes” on line 32 of the SVOC Data-Validation Checklist. • Contact the analytical laboratory and the SMO for the missing information. • If the laboratory is unable to provide the missing information, qualify all results as rejected (R, SV11) on the individual sample Form 1. • Go to Section 6.12, “Verifying the Dilutions.”

6.12 Verifying the Dilutions

IF the sample was...	THEN...
<u>Not</u> diluted,	<ul style="list-style-type: none"> • Record “No” on line 33 of the VOC Data-Validation Checklist. • Go to Section 6.13, “Identifying the Obvious Quality Deficiencies.”

IF the sample was...	THEN...
Diluted <u>and</u> no target analytes are detected above the second lowest standard,	<ul style="list-style-type: none"> • Record "Yes" on line 33 of the SVOC Data-Validation Checklist. • Contact the analytical laboratory and the SMO for the missing information. • If the laboratory cannot provide the missing information, qualify the affected samples as rejected (R, SV10) on the individual sample Form 1. • If the laboratory can provide proof of matrix interference that was not removed by acceptable cleanup attempts, qualify all the nondetected analytes as estimated (UJ, SV10) on the individual sample Form 1. • Go to Section 6.13, "Identifying the Obvious Quality Deficiencies."

6.13 Identifying the Obvious Quality Deficiencies

IF the validator...	THEN...
Does <u>not</u> notice any obvious data-quality deficiencies other than those covered by this SOP,	<ul style="list-style-type: none"> • Record "No" on line 34 of the SVOC Data-Validation Checklist. • Go to Section 6.14, Assembling and Submitting the Validation Data-Record Package."

IF the validator...	THEN...
<p>Notifies any significant or obvious data quality deficiencies during the data-validation process,</p>	<ul style="list-style-type: none"> • Record "Yes" on line 34 of the SVOC Data-Validation Checklist. • Contact the analytical laboratory and the SMO, if necessary, to resolve the quality issue. • Record the appropriate qualifiers to the data based on the validator's best professional judgment and apply reason code SV19. • Write a clear description of the quality issue that was flagged on the Data-Validation Cover Sheet. • Go to Section 6.14, "Assembling and Submitting the Validation Data-Record Package."

6.14 Assembling and Submitting the Validation Data-Record Package

1. Sign and date the completed Data-Validation Cover Sheet
2. Assemble the items listed below in the following order:
 - A. The completed Data-Validation Cover Sheet
 - B. The SVOC Data-Validation Checklist (completed in Sections 6.2 through 6.13)
 - C. Photocopies of the following:
 - Form (Form 1) on which data-validation qualifiers and reason codes were recorded (assemble in order by sample identification)
 - The data-record package chain of custody forms
2. Attach the data-validation record package with the original data package and submit to the FSF in accordance with LANL-RRES-R-SOP-15.09, "Chain of Custody for Analytical Data Packages."
3. Go to Section 6.15, "Lessons Learned."

7.0 LESSONS LEARNED

- 7.1 Before performing work described in this SOP, **RRES-R Personnel** should go to the Department of Energy Lessons Learned Information Services home page, located at <http://www.tis.eh.doe.gov/II/II.html>,

and/or to the LANL Lessons Learned Resources web page, located at http://www.lanl.gov/projects/lessons_learned/, and search for applicable lessons.

- 7.2 During work performance and/or after the completion of work activities, **RRES-R Personnel**, as appropriate, shall identify, document, and submit lessons learned in accordance with the LANL, Lessons Learned System located at http://www.lanl.gov/projects/lessons_learned/.

8.0 RECORDS

Although this SOP produces no records to submit to the Records Processing Facility (RPF) in the course of completing this procedure, the items identified in Section 6.14 are part of the data record package submitted to the RPF from the SMO in accordance with SOP-15.09.

9.0 REFERENCES

To properly implement this SOP, **RRES-R Personnel** should become familiar with the contents of the following documents located at http://erinternal.lanl.gov/home_links/Library_proc.shtml:

- EPA (US Environmental Protection Agency), "US EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," Publication 9240.1-05, EPA-540/R-94/012, Office of Solid Waste and Emergency Response, Washington, DC (October 1999).
- "Chain of Custody for Analytical Data Packages," Los Alamos National Laboratory document LANL-RRES-R-SOP-15.09.
- LANL (Los Alamos National Laboratory), "Environmental Restoration Project Statement of Work for Analytical Services," Los Alamos National Laboratory document RFP Number 9-SX1-Q4257, Revision 2 (July 1995).
- QP-2.2, Personnel Orientation and Training.
- QP-4.2, Standard Operating Procedure Development.

10.0 ATTACHMENTS

The **user** of this SOP may locate all forms associated with this procedure at <http://erinternal.lanl.gov/Quality/user/forms.asp>.

Attachment A: Semivolatile Organic Compounds Data-Validation Qualifier Flags, 1 page

Attachment B: Semivolatile Organic Compounds Data-Validation Reason Codes, 3 pages

Attachment C: Data-Validation Cover Sheet, 1 page

Attachment D: SVOC Data-Validation Checklist Form, 1 page

Attachment E: List of Acronyms and Abbreviations, 1 page

[Using a token card, click here to record "self-study" training to this procedure.](#)

If you do not possess a token card or encounter problems, contact the RRES-ECR training specialist.

Attachment A: Semivolatile Organic Compounds Data-Validation Qualifier Flags

- J The analyte is classified “detected,” but the reported concentration value is expected to be more uncertain than usual.
- J+ The analyte is classified “detected,” but the reported concentration value is expected to be more uncertain than usual with the potential for positive bias.
- J- The analyte is classified “detected,” but the reported concentration value is expected to be more uncertain than usual, with the potential for negative bias.
- U The analyte is classified “not detected.”
- UU The analyte is classified “not detected,” with an expectation that the reported result is more uncertain than usual.
- R The reported sample result is classified “rejected” because of serious noncompliance regarding quality-control acceptance criteria.

Attachment B: Semivolatile Organic Compounds Data-Validation Reason Codes

Code	Semi Volatiles	Qualifier Nondetects	Qualifier Detects	Description	Comments
0	SV0	R	J	The results/reporting limits for the affected analytes are considered estimated (J)/rejected (R) because the associated IS retention times have shifted by more than 30 s from the previous continuing calibration standard.	The validator must check the chromatogram profile to determine if any false positives or negatives exist. Reject (R) all analytes that are not present, and qualify as estimated (J) all analytes that are present with matching mass spectrums. If the mass spectrum does support the reported detection, consider the analyte undetected and reject the result (U, SV0).
1	SV1	UJ	J	The results/reporting limits for the affected analytes are considered estimated (J)/(UJ) because the associated IS area counts show less than 50%R or greater than 200%R when compared to the area counts in the applicable continuing calibration standard.	Use when the validator compares the CCV to the previous day's CCV. The validator qualifies only analytes related to failing ISs.
1a	SV1a	UJ	J	The results/reporting limits for the affected analytes are considered estimated (J)/(UJ) because the associated IS area counts are less than 50% but greater than 10%R when compared to the area counts in the applicable continuing calibration standard.	Use when the validator compares the CCV to the previous day's CCV. The validator qualifies only analytes related to failing ISs.
1b	SV1b	UJ	J	The results/reporting limits for affected analytes are considered estimated (J)/(UJ) because the associated IS area counts show greater than 200%R when compared to the area counts in the applicable continuing calibration standard. The validator qualifies only those analytes quantitated using the IS with >200%R.	
2	SV2	R	J	The results/reporting limits for affected analytes are considered estimated (J) for detected analytes /rejected (R) for nondetected analytes because the associated IS area counts show less than 10%R when compared to the area counts in the applicable continuing calibration standard.	
2a	SV2a	R	R	The required IS information is missing. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.
3	SV3	N/A	J+	The results for the affected analytes are considered estimated and biased high (J+) because 2 or more sample surrogate recoveries in the same fraction were greater than the UAL.	SVOCs must have at least two surrogates in one fraction out for qualification.
3a	SV3a	N/A	J-	The results for the affected analytes are considered estimated and biased low (J-) because at least two sample surrogate recoveries in the same fraction were less than the LAL but greater than 10%R.	SVOCs must have two surrogates in one fraction out for qualification. This code is used for detected analytes.
3b	SV3b	N/A	J-	The results for the affected analytes are considered estimated and biased low (J-) because at least one sample surrogate recovery was less than 10%R.	Code is used for detected analytes.

Code	Semi Volatiles	Qualifier Nondetects	Qualifier Detects	Description	Comments
3c	SV3c	UJ	N/A	The reporting limits for affected analytes are considered estimated (UJ) because at least two sample surrogate recoveries in the same fraction were less than the LAL but greater than 10%R.	SVOCs must have two surrogates in one fraction out for qualification. This code is used for nondetected analytes.
3d	SV3d	R	N/A	The reporting limits for affected analytes are considered rejected (R) because at least one sample surrogate recovery was less than 10%R.	This code is used for nondetected analytes.
3e	SV3e	UJ for LAL	J	The reporting limits/results for affected analytes in a fraction are considered estimated (UJ)/(J) because at least one sample surrogate %R is >UAL and at least one surrogate %R is < LAL.	SVOCs must have two surrogates in one fraction out for qualification. Qualify only those analytes in the affected fraction.
3f	SV3f	R	R	The required surrogate information is missing. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.
4	SV4	N/A	U	The results for the affected analytes are considered not detected (U) because the associated sample concentration was less than 5 times/10 times the amount in the method blank.	Effective dilutions must be considered for common laboratory contaminants.
4a	SV4a	N/A	J	The results for affected analytes are considered estimated (J) because the associated sample concentration was greater than 5 times/10 times the amount in the method blank.	
4b	SV4b	See comments	See comments	The required method blank or instrument blank documentation is missing. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.
7	SV7	R	R/J	The results for affected analytes are rejected (R) because the associated analyte did not have a valid 5-point calibration and/or a standard at the reporting limit. Detections within the valid range of calibration are qualified as estimated (J).	Qualify only the affected analytes. If the detection is between the lowest and highest acceptable standards, qualify as estimated (J).
7a	SV7a	UJ	J	The results/reporting limits for the affected analytes are considered estimated (J)/estimated (UJ) because the associated %RSD/%D exceeded the criteria in the initial or continuing calibration standards.	Qualify only the affected analytes.
7b	SV7b	R	J	The results/reporting limits for the affected analytes are considered estimated (J)/rejected (R) because the associated relative response factor (RRF) was less than 0.05.	Qualify only the affected analytes.
8	SV8	N/A	U	The results for the affected analytes are considered not detected (U) because the associated mass spectrum did not meet method specifications.	
8a	SV8a	See comments	See comments	The mass spectrum documentation is missing. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.
9	SV9	UJ	J-	The results/reporting limits for affected analytes are considered estimated and biased low (J-)/estimated (UJ) because the extraction holding time was exceeded by less than two times the published method for holding time.	
9a	SV9a	R	R	The results for the affected analytes are considered rejected (R) because the extraction holding time was exceeded by more than two times the published method for holding time.	

Code	Semi Volatiles	Qualifier Nondetects	Qualifier Detects	Description	Comments
9b	SV9b	R	R	The results for affected analytes are rejected (R) because the analytical holding time was exceeded.	
10	SV10	See comments	See comments	Undetected results for an affected analyte are considered estimated (UJ) or rejected (R) because the laboratory diluted the sample for matrix interferences.	Qualify all results as rejected if the laboratory cannot provide proof of cleanup or matrix interferences. Qualify nondetected results as estimated if the laboratory can provide evidence of cleanup and/or matrix interferences not subject to acceptable cleanup methods.
12	SV12	R	R	The LCS documentation is missing. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.
12a	SV12a	R	J-	The results/reporting limits for affected analytes should be regarded as estimated and biased low (J-)/rejected (R) because the associated LCS recovery was less than 10%R.	If less than a full list of LCSs is provided, use professional judgment to qualify all analytes in that fraction (SVOC) and/or all analytes quantitated by the same IS as the affected analyte.
12b	SV12b	N/A	J-	The results for the affected analyte are considered estimated and biased low (J-) because the associated LCS recovery was < LAL but greater than 10%R.	If less than a full list of LCSs is provided, use professional judgment to qualify all analytes in that fraction (SVOC) and/or all analytes quantitated by the same IS as the affected analyte. This code is for nondetected analytes.
12c	SV12c	UJ	N/A	The reporting limits for an affected analyte are considered estimated (UJ) because the associated LCS recovery was < LAL but greater than 10%R.	If less than a full list of LCSs is provided, use professional judgment to qualify all analytes in that fraction (SVOC) and/or all analytes quantitated by the same IS as the affected analyte. This code is for nondetected analytes.
12d	SV12d	N/A	J+	The results for an affected analyte are considered estimated and biased high (J+) because the associated LCS recovery was > UAL.	If less than a full list of LCSs is provided, use professional judgment to qualify all analytes in that fraction (SVOC) and/or all analytes quantitated by the same IS as the affected analyte.
15	SV15			The results for the affected sample could not be analyzed because of insufficient sample volume or problems at the analytical laboratory.	
16	SV16	See comments	See comments	The required calibration information is missing or samples were analyzed on an expired calibration. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.
16a	SV16a	R	R	The results/reporting limits for the affected analytes are rejected because the instrument performance (DFTPP) sample did not pass method acceptance criteria.	
19	SV19	See comments	See comments	The validator identified quality deficiencies in the reported data that require qualification. See the Data-Validation Cover Sheet for specific details.	Apply the qualifier appropriate to identify the effect of the quality deficiency on the reported data.

Note: The validator must notify the project manager and the SMO of any rejected data for potential nonpayment.

Attachment C. Data-Validation Cover Sheet

☐

Rejected Data

Section I

Request Number:

Validation Date:

Lab Code:

Contract Laboratory Name:

Validator:

Organization:

Analytical Suite (check all that apply):

☐ Volatile Organics

☐ Semivolatile Organics

☐ Organochlorine Pesticides/Polychlorinated Biphenyls

☐ High Explosives

☐ Inorganics

☐ Radiochemistry

Other (describe):

Section II—Completeness Check

Yes	No	n/a	(check one)
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	1. Chain-of-custody form(s)
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	2. Case narrative
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	3. Sample result form(s)
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	4. Sample chromatograms
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	5. Standard chromatograms

Yes	No	n/a	(check one)
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	6. Raw/BSS data
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	7. Quality control forms
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	8. Quantitation reports
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	9. TICs forms
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	10. TICs mass spectra

Comments/problems noted (include information about requests for further information submitted to the contract laboratory and agreed-upon date of resolution and contract laboratory point of contact):

(Attach additional comment sheets as necessary.)

Validator's signature:

Date:

SOP-15.02, R1

**Los Alamos National Laboratory
RRES-Remediation Program**

Attachment D: SVOC Data-Validation Checklist

Yes	No	(check one)	Assign qualifier listed below if criteria = Yes	
			Detected analyte	Undetected analyte
<input type="checkbox"/>	<input type="checkbox"/>	1. The Sample was extracted >1 to =2 times the appropriate hold time requirement.	J-, SV9	UJ, SV9
<input type="checkbox"/>	<input type="checkbox"/>	2. The Sample was extracted >2 times the appropriate holding time requirement.	R, SV9a	R, SV9a
<input type="checkbox"/>	<input type="checkbox"/>	3. The Sample was analyzed outside the analytical holding time requirement.	R, SV9b	R, SV9b
<input type="checkbox"/>	<input type="checkbox"/>	4. The instrument performance check (DFTPP) is not present or fails acceptance criteria.	R, SV16A	R, SV16A
<input type="checkbox"/>	<input type="checkbox"/>	5. The initial calibration is not present.	R, SV16	R, SV16
<input type="checkbox"/>	<input type="checkbox"/>	6. The initial calibration does not have either 5 calibration points or a low standard at the reporting limit.	R, SV7	R, SV7
<input type="checkbox"/>	<input type="checkbox"/>	7. The initial calibration analyte %RSD is >30%.	J, SV7a	UJ, SV7a
<input type="checkbox"/>	<input type="checkbox"/>	8. The analyte minimum RRF is <0.05 in the initial calibration.	J, SV7b	R, SV7b
<input type="checkbox"/>	<input type="checkbox"/>	9. CCV is not present.	R, SV16	R, SV16
<input type="checkbox"/>	<input type="checkbox"/>	10. The continuing calibration analyte %D is >25%.	J, SV7a	UJ, SV7a
<input type="checkbox"/>	<input type="checkbox"/>	11. The analyte minimum RRF is <0.05 in the continuing calibration.	J, SV7b	R, SV7b
<input type="checkbox"/>	<input type="checkbox"/>	12. The method blank information is not present.	R, SV4b	R, SV4b
<input type="checkbox"/>	<input type="checkbox"/>	13. The analyte detected in the method blank and sample result for analyte is =5x/10x the amount in blank.	U, SV4	N/A
<input type="checkbox"/>	<input type="checkbox"/>	14. The analyte detected in the method blank and sample result for analyte is >5x/10x the amount in method blank	J, SV4a	N/A
<input type="checkbox"/>	<input type="checkbox"/>	15. IS information is not present.	R, SV2a	R, SV2a
<input type="checkbox"/>	<input type="checkbox"/>	16. The IS area in the CCV is <50%R or >200%R.	J, SV1	UJ, SV1
<input type="checkbox"/>	<input type="checkbox"/>	17. The IS retention times have shifted by more 30 s.	J, SV0	R, SV0
<input type="checkbox"/>	<input type="checkbox"/>	18. The sample IS area is >200%R.	J, SV1b	N/A
<input type="checkbox"/>	<input type="checkbox"/>	19. The sample IS area is <50%R but =10%R. The s	J, SV1a	UJ, SV1a
<input type="checkbox"/>	<input type="checkbox"/>	20. The Sample IS area is <10%R.	J, SV2	R, SV2
<input type="checkbox"/>	<input type="checkbox"/>	21. The surrogate information is not present.	R, SV3f	R, SV3f
<input type="checkbox"/>	<input type="checkbox"/>	22. The surrogate %R is greater than the UAL.	J+, SV3	N/A
<input type="checkbox"/>	<input type="checkbox"/>	23. The surrogate %R is greater than the LAL but =10%.	J-, SV3a	UJ, SV3c
<input type="checkbox"/>	<input type="checkbox"/>	24. The surrogate %R is <10%.	J-, SV3b	R, SV3d
<input type="checkbox"/>	<input type="checkbox"/>	25. There is one surrogate %R <LAL and one surrogate %R >UAL.	J, SV3e	UJ, SV3e
<input type="checkbox"/>	<input type="checkbox"/>	26. LCS information is not present.	R, SV12	R, SV12
<input type="checkbox"/>	<input type="checkbox"/>	27. The LCS %R is >UAL.	J+, SV12d	N/A
<input type="checkbox"/>	<input type="checkbox"/>	28. The LCS %R is <LAL but =10%.	J-, SV12b	UJ, SV12c
<input type="checkbox"/>	<input type="checkbox"/>	29. The LCS %R is <10%	J-, SV12a	R, SV12a
<input type="checkbox"/>	<input type="checkbox"/>	30. Mass spectral information is missing.	R, SV8a	R, SV8a
<input type="checkbox"/>	<input type="checkbox"/>	31. The mass spectrum does not meet method specifications.	U, SV8	N/A
<input type="checkbox"/>	<input type="checkbox"/>	32. TICs were requested but not reported.	none	none
<input type="checkbox"/>	<input type="checkbox"/>	33. The sample was diluted inappropriately.	N/A	UJ, V10
<input type="checkbox"/>	<input type="checkbox"/>	34. Other obvious data quality issues were identified.	__, SV19	__, SV19

SOP-15.02, R1

**Los Alamos National Laboratory
RRES-Remediation Program**

Attachment E: List of Acronyms and Abbreviations

BSS	background subtracted spectra	%D	percent difference
CCV	continuing calibration verification	%R	percent recovery
CLP	contract laboratory program	%RSD	percent relative standard
COC	chain of custody	QC	quality control
DFTPP	decfluorotriphenylphosphine	QP	quality procedure
EPA	U.S. Environmental Protection Agency	RICs	reconstructed ion chromatograms
EQL	estimated quantitation limit	RFP	Records Processing Facility
RRES-R	environmental restoration	RL	reporting limit
FSF	Field Support Facility	RN	request number
GC/MS	gas chromatography mass spectrometry	RPF	Records Processing Facility
HEs	high explosives	RRF	relative response factor
IS	internal standard	RT	retention time
LAL	lower acceptance limit	SMO	Sample Management Office
LANL	Los Alamos National Laboratory	SOP	standard operating procedure
LCS	laboratory control sample	SOW	statement of work
MDL	method detection limit	SVOC	semivolatile organic compound
n/a	not analyzed	TIC	tentatively identified compound
		UAL	upper acceptance limit